

Integrating Theory and Practice: An Experimental Pedagogy for Teaching Crude Oil Flow Properties

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Abstract: To further enhance the practical experimental skills of undergraduate students majoring in applied chemistry, a comprehensive experiment was developed focusing on crude oil viscosity, yield stress, viscosity-temperature behavior, and the evaluation of viscosity-reducing agents. By measuring various physical parameters of crude oil and assessing the performance of viscosity reducers, students gain a thorough understanding of crude oil characteristics and the effectiveness of flow improvers. This experimental design is highly targeted and practically relevant, effectively reinforcing students' comprehension of key physical properties of crude oil while strengthening their abilities in experimental operation, observation, and problem-solving. Moreover, the experiment not only consolidates foundational knowledge but also provides systematic training in literature research, experimental design, hands-on execution, data analysis, and the preparation of comprehensive laboratory reports.

1. Introduction

The global demand for oil continues to rise as industries worldwide expand. Following more than a century of extraction, conventional crude oil reserves have been steadily declining. Therefore, the extraction heavy crude oil, which is relatively abundant in reserves, represents a significant strategy for mitigating resource scarcity. Understanding the physical properties of crude oil is crucial for the effective exploitation and utilization of this resource [1]. In the practical courses for undergraduates, introducing experimental teaching content (such as the determination of crude oil physical property parameters: relative density, pour point, and viscosity of crude oil), combining basic theories, professional knowledge, and practical operations, helps students to systematically and comprehensively understand crude oil extraction and utilization knowledge, while enhancing their

ability to analyze and solve practical engineering problems.

The relative density of crude oil significantly influences the costs associated with its extraction, storage, transportation, and processing, and directly affects the corresponding economic returns [2]. The pour point and viscosity of crude oil are two essential parameters for assessing its physical properties. The measurement of these properties is fundamental to understanding the rheological behavior of crude oil, which has strategic implications for enhancing operational efficiency and economic viability across the entire crude oil value chain [3-6]. And the pour point of crude oil refers to the temperature at which the oil transitions from a fluid state to a loss of flowability during cooling, and it is closely related to the wax content present in the crude oil. During pipeline transportation of waxy crude oil, a temperature drops below the pour point triggers the gradual precipitation of wax crystals, which interact with resins and asphaltenes present in the crude oil. This interaction leads to the formation of a three-dimensional network structure, ultimately resulting in the loss of fluidity of the crude oil as a whole. The yield value characterizes waxy crude oil by quantifying the minimum shear stress that must be applied under set conditions to overcome its pre-existing wax crystal structure and induce flow [7].

Crude oil viscosity is defined as the ratio of shear stress to the velocity gradient per unit area at any given point in the fluid, representing its internal resistance to flow. Higher viscosity indicates greater resistance to flow and, consequently, more difficulty in fluid movement. In practical applications, the viscosity-temperature curve is frequently utilized to reflect the variation of crude oil viscosity with temperature, which holds significant importance for the design, production and research related to crude oil gathering, transportation, and storage [8]. Compared with conventional oil and gas resources, China possesses abundant reserves of heavy oil. Heavy oil refers to crude oil with a higher density or viscosity than conventional oil under normal reservoir conditions [9]. Therefore, viscosity reducers are commonly added during extraction and transportation (as shown in Figure 1) to improve the viscosity and flowability of crude oil [10], and the effectiveness of the reducers is evaluated through viscosity-temperature curves [11].

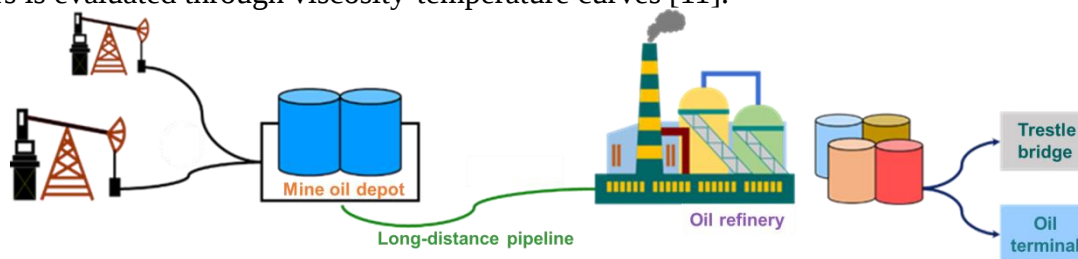


Figure 1: Schematic diagram of crude oil gathering and transportation

Measuring the physical properties of crude oil constitutes essential professional knowledge that applied chemistry students in petroleum-focused institutions must acquire and master. This experiment demands a comprehensive grasp of both theoretical principles and procedural steps, fostering the development of critical skills such as independent thinking, analytical reasoning, practical experimentation, data interpretation, and problem-solving [12]. The experimental setup is designed to be completed within four instructional periods, encompassing the measurement of key crude oil parameters and the assessment of viscosity reducer performance, while simultaneously strengthening students' theoretical understanding through direct engagement with laboratory instruments and hands-on practice. The design of this experiment aims not only to reinforce students' foundational knowledge, but also to provide systematic training in their abilities to retrieve literature, design experiments, conduct experimental operations, organize data, and compose comprehensive experimental reports.

2. Objectives of the Experiment

- (1) Students can gain a fundamental understanding of the rheological behavior and viscosity characteristics of crude oil;
- (2) Students can acquire proficiency in experimental techniques for measuring crude oil viscosity and yield stress;
- (3) Students can develop the ability to construct and interpret viscosity-temperature profiles;
- (4) Students are able to familiarize themselves with the selection criteria and performance evaluation procedures for viscosity reducing additives.

3. Experimental Principles

3.1 The Source of Viscosity

Crude oil consists of four main fractions: saturated hydrocarbons, aromatic hydrocarbons, resins, and asphaltenes. Among them, the content of resins and asphaltenes is the main influencing factor for the high viscosity of heavy oil, and resins could dissolve and disperse asphaltenes. When the temperature or pressure drops, the asphaltenes in crude oil interact and aggregate, forming a porous matrix that could entrap liquid hydrocarbons and significantly increase the viscosity of the crude oil. At the same time, the resins in the crude oil precipitate and adsorb onto the surface of the network structure to form a solvation layer. As the temperature drops, the thickness of the solvation layer increases and the intermolecular forces intensify, resulting in an increase in viscosity [4, 13].

3.2 Measurement of Viscosity

For a given shear rate, the viscosity of a sample increases linearly with the applied shear stress. In laboratory settings, rotational viscometers are commonly employed to measure the viscosity of crude oil. Their core design features a stationary component and a rotating element, the latter of which is termed the rotor. As the rotor rotates through the sample, it experiences resistance, which in turn transmits a corresponding torque to the drive shaft and motor. This torque can be measured using an electronic sensor, allowing for the determination of the sample's viscosity [11]. It can be expressed by the formula (1):

$$\eta = \frac{\tau}{\dot{\gamma}} = \frac{Z \cdot a}{\dot{\gamma}} \quad (1)$$

Where η represents the dynamic viscosity (Pa s); τ represents the shear stress (Pa); $\dot{\gamma}$ represents the shear rate (s^{-1}); Z represents the instrument system constant (Pa); a represents the instrument reading value.

3.3 Determination of Yield Value

When crude oil cools below the wax precipitation point, wax crystals begin to precipitate and progressively develop a structurally stable network exhibiting measurable mechanical strength. Rotational viscometers are also used in laboratories to determine the yield value of crude oil. The oil sample is placed into the rotational viscometer's cylindrical measuring system and cooled at a controlled constant rate to the target temperature. Following a sufficient period of thermal stabilization, the sample loses its flowability. Subsequently, a defined shear rate is applied, and the critical shear stress at which the rotor begins to rotate continuously is recorded. This measured stress is designated as the yield value of the oil sample under the specified testing conditions. Based on the operating principle of the rotational viscometer, the yield value could be determined using the

following formula (2) [7, 14]:

$$\tau_y = Z \cdot \alpha \quad (2)$$

Where τ_y is the crude oil's yield value under the specified testing condition (Pa); Z represents the system's instrumental constant; α is the instrument reading (Pa).

3.4 The Viscosity-Temperature Curve

To quantify the temperature-dependent flow behavior, rotational viscosity measurements were conducted on the crude oil specimens at a series of given temperatures. And the experimental viscosity-temperature relationship was graphically represented on semi-logarithmic coordinates, and data points exhibiting significant deviation were subsequently identified [15].

3.5 Assessment of Viscosity Reducer

The viscosity reducer was loaded into the crude oil as required, and its effectiveness was assessed by measuring the oil's viscosity and pour point.

3.6 Instruments and Chemical Regents

Cylindrical container, hydrometer, constant-temperature water bath and oil bath, plastic or glass stirring rod, round-bottomed glass test tubes, round-bottomed glass sleeves (Figure 2a), mercury thermometers and ordinary thermometers (range: -30 °C~20 °C and -30 °C~100 °C, division value of 1 °C), cooling bathes, refrigeration equipment, grinding bottles, rotational viscometer (Figure 2b), measuring cylinder (D100/300), high-pressure storage tank, high-pressure constant-speed pump, electronic balance, beaker (1 L, 500 mL, 250 mL), semi-logarithmic coordinate paper, ruler, pencil, crude oil sample and viscosity reducer.

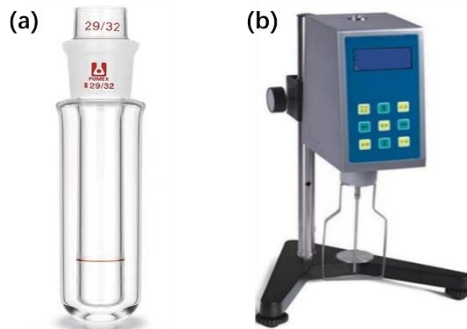


Figure 2: Instruments used in the experiments: (a) pour point test glass sleeve (b) rotational viscometer

4. Experiment content and Procedure

4.1 Pre-treatment of Crude Oils

The oils should initially be filtered, followed by dehydration. During filtration, a filter screen with a pore size of 0.043 mm should be used, and the filtration temperature should be maintained below 100 °C. For the dehydration process, either high-temperature treatment or centrifugal dehydration may be employed, with the heating temperature kept below 120 °C. The final moisture content of the oil sample should be reduced to less than 0.5%. The physical parameters (including the viscosity, yield value, and wax precipitation point) of crude oils were determined by using the rotational

viscometer.

4.2 Measurement of Density

(1) Introduce the oil specimen into a clean graduated cylinder that had been preheated to the target temperature, ensuring minimal turbulence, aeration and evaporation of light hydrocarbons [16].

(2) Eliminate the surface bubbles from the oil specimen via a clean filter paper.

(3) Position the prepared cylinder containing the oil specimen upright in an area free of air flow. The ambient temperature should remain stable, with fluctuations not exceeding 2 °C throughout the testing period. Maintain the sample in a thermostatically-controlled water bath if ambient thermal variations exceed 2 °C.

(4) Stir the oil sample vertically using a glass rod or a thermometer. If a resistance thermometer is employed, use a stirring rod to ensure uniform density and temperature throughout the cylinder. Once the temperature change remains within 0.1 °C, remove the glass rod or the thermometer from the graduated cylinder.

(5) Place the hydrometer in the crude oil sample to the equilibrium position and release it. Ensure that the hydrometer remains buoyant in the test oil and prevent any liquid from wetting the upper portion of the main tube. Depress the hydrometer 1-2 mm to the equilibrium line, then release it and allow it to stabilize freely. Observe the curvature of the liquid-air interface. If the meniscus moves, clean the main tube of the hydrometer. Continue the process until the meniscus profile stabilizes and remains unchanging.

(6) After recording the hydrometer reading, quickly and carefully remove the hydrometer. Agitate the test oil perpendicularly with a thermometer and record the thermal reading to the nearest 0.1 °C. If the temperature deviates from the initial test temperature by more than 0.5 °C, repeat the temperature and hydrometer readings until the variation remains within ± 0.5 °C. If a stable temperature could not be achieved, immerse the thermometer and measurement apparatus containing the test oil in a thermostatically-controlled water bath and re-calibrated the readings.

(7) The lead elastic-sealed densitometer should be air-dried and cooled vertically after use at temperatures exceeding 38 °C.

4.3 Determination of Pour Point

(1) Place the container with the oil sample into a pre-heated water bath. Then transfer the sample into multiple pour point test glass sleeves and place the sleeves in the water bath to heat to 50 ± 1 °C. If the preheating temperature requirement exceeds 50 °C, after heating to the specified preheating temperature in the water bath, activate the refrigeration instrument or use cool water to gradually reduce the temperature of the specimen to 50 ± 1 °C. In certain cases, preheating may be omitted depending on the specific conditions [17].

(2) Place the thermometer-equipped cork into the mouth of the test glass sleeves, center the thermometer within the vessel, and keep the mercury bulb at a distance of 20 ± 2 mm from the base of the vessel. Subsequently, detach and temporarily set aside the sealing stopper.

(3) Heat the required test glass sleeves to 50 ± 1 °C. If the crude oil sample temperature is lower than that of the sleeves, heat the sample to match the sleeve temperature. Prepare the coolant and estimate the pour point of the specimen in advance. Prepare a series of thermostatted baths, with the first bath maintained at 25 ± 2 °C, and gradually decrease the temperature by 15 °C to maintain a cooling rate of 0.5~1.0 °C/min for the oil sample. Install the tubing vertically in the cooling bath, ensuring that the immersion depth is no less than 100 mm.

(4) Introduce the test oil to the calibration mark on the glass sleeve. Promptly insert the thermometric assembly centrally into the vessel and transfer the prepared sleeve smoothly into the

cooling bath. Ensure an initial thermal differential of 10–25 °C between the specimen and coolant. If the differential falls below 10 °C prior to solidification, proceed to the subsequent cooling stage without agitating the sample.

(5) Once the thermal reading exceeds the projected value by 8 °C, remove the test tube from the glass sleeve casing and gently incline it to assess for any evidence of fluid meniscus displacement. If movement is observed, quickly and steadily reinsert the test tube into the casing for further cooling. The total time for removing and reinserting the test tube should not exceed 3 seconds. Thereafter, observe the oil sample every time the temperature drops by 2 °C until the liquid level ceases to move. Then, rapidly lay the test tube flat for 5 seconds. If the liquid level remains stationary, record this temperature as the pour point. Upon detection of meniscal displacement, immediately reorient the test vessel vertically into the bath and resume cooling. Continue repeating the above procedure until there is no further movement of liquid surface, and record the corresponding pour point. If the pour point cannot be determined under these conditions, substitute the oil sample and reduce the anticipated pour point by 4 °C prior to repeating the procedure [17].

4.4 Measurement of Viscosity

(1) After processing, the oil specimen is transferred into the high-pressure storage tank. Subsequently, high-pressure constant-speed pump is used to transfer the oil sample from the high-pressure storage tank into the D100/300 measuring cylinder until the oil specimen partially spills over the top of the cylinder. When using an open-cup-type measuring cylinder for sample loading, the treated oil sample shall be carefully poured along the inner surface of the cup until it reaches the specified mark [18].

(2) Connect the power supply and sequentially activate the power switches of the instruments. Subsequently, input the name and number of the crude oil sample, select the appropriate measurement probe and rotor, and then establish the target shear conditions and temperature setpoint.

(3) The temperature of the oil bath should be preset to ensure thermal stability. Once a stable temperature has been achieved, each sample shall be introduced into the oil bath and equilibrated for a period of 20 to 40 minutes. Viscosity measurements shall be conducted on each oil sample at no fewer than six discrete temperature points. The measurement sequence shall proceed incrementally from the lowest to the highest temperature. Prior to each measurement, a zero-point calibration shall be executed to ensure measurement accuracy.

(4) At the selected shear rate, once the instrument reading has achieved initial stabilization, record the instrument reading value “a”. Subsequently, record the reading at five-minute intervals. If the arithmetic mean of the last three values from a set of four consecutive measurements deviates from the preceding value by no more than 5%, it is determined that the equilibrium state has been reached, and the measurement of the value “a” at the current shear rate is considered complete. In the case of non-newtonian fluids, a fresh sample shall be employed when altering the test temperature [18].

(5) The viscosity of the crude oil sample could be calculated using formula (1).

4.5 Determination of Wax Precipitation Point

(1) Preheat the crude oil specimen 10-15 °C above its pour point, and load the oil as required by the instrument measurement system. Then activate the power switches of the constant-temperature circulator water bath and the measurement equipment, heat the oil specimen and the measurement system to 70-80 °C, and hold for 10 minutes [19].

(2) Set the thermostatic bath to maintain a continuous cooling ramp at a rate of 0.2-1 °C/min, while starting the rotational viscometer to operate at a certain fixed shear rate within the range of 10-600 s⁻¹.

(3) Acquire paired data of rheological parameters (shear stress or viscosity) and temperature at 1 °C decremental intervals. Construct semi-logarithmic plots in real-time using integrated software. Upon detection of a substantial slope transition in the curve, continue to measure data from 5 to 7 different temperature points [18].

4.6 Determination of Yield value

(1) Connect the power supply to the constant-temperature water bath, switch on the device, and set the temperature to 15-20 °C higher than the crude oil's pour point. Subsequently, preheat the oil specimen in the constant-temperature water bath, and select an appropriate rotor for the rotational viscometer based on the characteristics of the oil sample [19].

(2) Based on the density of crude oil, take a sample equivalent to the determined volume and transfer it into the preheated outer cylinder for analysis. For samples that remain fluid at ambient temperature, load the sample directly in accordance with the specified volume. Regulate the constant temperature circulator to achieve a controlled static cooling to the designated test temperature at a cooling rate ranging from 0.3 °C/min to 0.5 °C/min, followed by maintaining the temperature for a duration of 60 minutes.

(3) For rotational viscometers equipped with manually adjustable shear rate controls, select the lowest available shear rate. For systems equipped with continuous adjustment, set the shear rate to 0.2 s⁻¹. Initiate operation of the rotational viscometer and record the instrument reading (denoted as "α") or the corresponding shear stress value at the onset of rotation of the cylindrical component.

(4) The crude oil's yield value could be calculated using formula (2).

4.7 Evaluation of Viscosity Reducers

(1) Based on the designated concentration, introduce the viscosity reducer into multiple graduated cylinders containing 25 ml of oil, to prepare the diluted viscosity reducer "A".

(2) Introduce the diluted viscosity reducer "A" to the crude oil specimen to produce the experimental oil mixtures with viscosity reducer concentrations of 0, 100, 300, 500, 700, and 900 mg/L, respectively. Then, heat the oil samples to 50 °C while stirring them thoroughly.

(3) Determine the viscosity of oil samples from near the its pour point to 70 °C in accordance with Sections 4.3 and 4.4.

5. Results and Discussion

Based on the calculated viscosity values (vertical axis) and their corresponding temperature data (horizontal axis), construct the viscosity-temperature curve and mark the anomalous points on the horizontal coordinate. Connect the data points representing the temperature range of non-Newtonian fluid behavior using smooth curves, and annotate the corresponding shear rates associated with each curve. Then describe viscosity-temperature behavior of the oil sample across different temperature conditions.

Construct the viscosity-temperature curves for oil samples at varying concentrations of viscosity reducers, and compare these with the blank oil sample to evaluate the viscosity reduction performance and identify the optimal dosage. Additionally, compare the freezing point of the oil sample before and after the addition of the viscosity reducer.

6. Post-Class Reflective Inquiry

(1) In combination with knowledge of oilfield chemistry, consider what factors affect the pour

point and viscosity of crude oil?

(2) Review the relevant literature to gain an understanding of the mechanisms through which viscosity reducers function to decrease viscosity.

(3) Furthermore, what critical factors should be considered during the extraction, storage, and transportation of high-viscosity and high-coagulation crude oil?

7. Conclusion

This experimental design is based on the fundamental principles of "Oilfield Chemistry," with the aim of applying theoretical knowledge through the measurement of crude oil's physical parameters and the evaluation of viscosity-reducing agents. It enhances students' practical operational skills and observational abilities while deepening their understanding of integrated oilfield chemistry experiments and reinforcing key theoretical concepts. In addition to requiring students to follow established protocols and demonstrate hands-on proficiency, the experiment emphasizes the analysis of various stages and outcomes to identify patterns, detect anomalies, and formulate solutions. By integrating theoretical knowledge with practical application, the experiment effectively fosters the development of innovative thinking.

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